



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	Stephen D. Pacetti	Examiner:	Loan H. Thanh
Serial No.	09/406,473	Art Unit:	3763
Filed:	September 27, 1999		
Title:	Drug Diffusion Barriers For A Catheter Assembly		

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Commissioner for Patents

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APPEAL BRIEF

Sir:

On December 4, 2006, applicant appealed to the Board of Patent Appeals from the nonfinal rejection of Claims 139-154 which have been twice rejected. The following is applicant's Appeal Brief submitted pursuant to 37 C.F.R. §41.37.

Real Party in Interest

This application is assigned to Advanced Cardiovascular Systems, Inc. which is now Abbott Vascular, a division of Abbott of Abbott Park, Illinois, which is the real party in interest in this appeal.

Related Appeals and Interferences

Applicant and his counsel are not aware of any related appeals or interferences which would affect, be affected by, or have a bearing on the instant appeal.

Status of Claims

Claims 139-154 are pending and have been twice rejected by the Examiner. Claims 142, 143, and 147 form the subject of this appeal.

Claims 1-138 have been canceled.

Status of Amendments

There are no unentered amendments.

Summary of Claimed Subject Matter

The pending claims include one independent claim: Claim 139.

Applicant's invention recited in independent claim 139 is directed to a sheath (22) comprising a hollow body, the sheath (22) being adapted to removably cover at least part of an implantable medical object (12 and 16) that carries a therapeutic substance, wherein the sheath material prevents the therapeutic substance from significantly absorbing into the sheath (22) (Figure 1 and the specification, page 6, line 26 to page 8, line 17), wherein the sheath material has an oxygen transmission rate of not more than 200 cc/100 in² for 1 mil per 24 hours at 73°F, 75% relative humidity and 1 atmosphere, and wherein the sheath material has a water vapor transmission rate of not more than 20 gm/100 in² for 1 mil per 24 hrs. 100°F 90% relative humidity and 1 atm (specification, page 9, lines 16-20). As recited in dependent Claim 140, the implantable object (12 and 16) is a balloon (16) integrated with a catheter (12) (specification, page 7, line 8). Claim 141 recites that the balloon material, sheath material, or both comprise the same or different polymeric material (specification, page 9, lines 13-15). Claim 142 recites that the polymeric material comprises a polyurethane having a glass transition temperature above a storage temperature (specification, page 10, lines 23-24). Claim 143 recites that the polymeric material comprises a polyurethane having a non-polar soft segment wherein the non-polar soft segment is selected from hydrocarbons, silicones, fluorosilicones, or their mixtures (specification, page 10, lines 25-27).

Grounds of Rejection to Be Reviewed on Appeal

Issue 1: Whether Claim 142 is obvious over Sanhatjian et al. (U.S. Patent 5,674,192) ("Sanhatjian") under 35 U.S.C. § 103.

Issue 2: Whether Claim 143 is obvious over Sanhatjian under 35 U.S.C. § 103.

Issue 3: Whether Claim 147 is obvious over Sanhatjian under 35 U.S.C. § 103.

Argument

Issue 1

The Examiner has rejected Claim 142 as being obvious over Sanhatjian. The rejection is improper because the cited art does not disclose at least the following limitation of Claim 142: “the polymeric material comprises a polyurethane having a glass transition temperature above a storage temperature” and because there is no motivation or suggestion in the record to modify the cited art to include the limitation.

The record shows that the Examiner takes the position (Office Action mailed September 7, 2006 and Office Actions going back to October 5, 2004) that it is inherent that the polyurethane has a glass transition temperature (T_g) that is above the storage temperature. The inherency theory is based on two grounds: (1) that it is inherent that the polyurethane of Sanhatjian has a glass transition temperature (T_g) that is above storage temperature because the storage temperature can be “any temperature” and (2) it would be inherent “for the T_g to be above storage temperature since the sheath would be a solid structure” and that “if the T_g were below the storage temperature then the sheath would be a liquid or unstable form during storage.” These two grounds are without merit.

With respect to the first ground of inherency, the Examiner asserts that “in the broadest interpretation the storage temperature can be any temperature.” The Examiner’s interpretation of the “storage temperature can be any temperature” is entirely inconsistent with the MPEP and with Federal Circuit Case law. The MPEP § 2111 directs the Examiner to give the claims their broadest reasonable interpretation consistent with the specification. MPEP § 2111 also states that “the broadest reasonable interpretation of the claims must also be consistent with the interpretation that those skilled in the art would reach.” In re Cortright, 165 F.3d 1353, 1359 (Fed. Cir. 1999) Therefore, the Examiner must view the claim terms through the eyes of a skilled artisan and consider the specification. Applicant submits that a skilled artisan knows how to pick reasonable storage temperatures for medical devices based on the devices’ constituency and intended use.

Based on this correct interpretation of storage temperature, the limitation of Claim 142 is not inherent in Sanhatjian. Sanhatjian discloses a catheter comprising a sheath

extendable over a balloon to inhibit release of drug from a coating over an expandable portion of the catheter. (Col. 3, lines 23-25, Sanhatjian) Furthermore, it is disclosed that the sheath can be made of polyurethane (for example, Tecoflex™) or TEFLON (fluorinated polymer). (Col. 6, lines 62-63, Sanhatjian) The Evidence Appendix provides a copy of *An investigation of the Fatigue Induced Failure Modes of Fiber/Elastomer Composites as Bearing Surfaces in Total Hip Joint Prosthesis*, published by the National Textile Center in its 1997 Annual report. This article was submitted with the response filed January 5, 2005 and shows the measurement of some Tecoflex™ polyurethanes. Reference to Table 1 of the Appendix shows that a medical grade Tecoflex™ has a Tg as low as -69.10, which is well below a reasonable storage temperature.

According to MPEP § 2112, inherency may not be established by probabilities or possibilities.” (quoting In re Robertson, 169 F.3d 743, 745, 49 USPQ2d 1949, 1950-51 (Fed. Cir. 1999). “In relying upon the theory of inherency, the examiner must provide a basis in fact and/or technical reasoning to reasonably support the determination that the allegedly inherent characteristic necessarily flows from the teaching of the applied prior art.” MPEP § 2112 (quoting Ex Parte Levy, 17 USPQ2d 1461, 1464 (Bd. Pat. App. & Inter. 1990)).

Since the allegedly inherent characteristic must flow from the prior art teaching, even a single example on the record contrary to the Examiner’s inherency theory precludes finding that a characteristic is inherent in the prior art. Thus, since extrinsic evidence in the above-mentioned article shows that the Tg of polyurethane is not necessarily above a reasonable storage temperature, Claim 142 is not inherent.

With respect to the Examiner’s second ground of inherency, the record shows that the Examiner makes certain factual assumptions in the Office Action mailed September 7, 2006: (1) the sheath must remain a solid to function; and (2) that if Tg is below a storage temperature, the material will either not be a solid or will be unstable. While the sheath’s function may require that the sheath remain a solid, nothing in its function requires that the sheath remain rigid. Thus, there is nothing in the record to support (2). A Tg below a storage temperature is perfectly compatible with the object being a solid. Tg is not a melting temperature of a polymer. Tg is the point at which the polymer begins the transition from behaving in a crystalline manner to behaving in an elastomeric manner. The transition from

crystalline to elastomeric occurs over a broad temperature range wherein Tg is somewhat arbitrarily placed.

Many polymers have a Tg below room temperature or a reasonable storage temperature, yet they remain solids. These facts are well known to a person of skill in the art. Medical polymers such as Tecoflex™ and common rubbers have a Tg below room temperature and body temperature and yet remain solid above their respective Tg's. If they did not, such polymers would not be used in medical applications or automobile tires under conditions above their respective Tg's. In addition, a Tg below a storage temperature is perfectly compatible with an object being stable. Tecoflex™ and rubber would not be used for medical applications and tires, respectively, at temperatures above their respective Tg's if they were unstable at these temperatures. Thus, since extrinsic evidence shows that a polymer with a Tg below a storage temperature can be a solid or stable, it is not inherent that the polyurethane has a Tg that is above storage temperature.

Furthermore, the record shows that the Examiner takes the position that skilled artisans would immediately recognize that properties such as flexibility and bendability (properties indicative of a material tending to be above its Tg) are useful in the medical arts (Office Action mailed September 7, 2006, page 6, lines 4-6), but also takes the position that keeping the polymeric material solid, with the material's storage temperature below Tg, is inherent in prior art polymers. These two positions are diametrically opposed.

Applicant has established that the recited limitations of Claim 142 are not inherent. Furthermore, the Examiner has provided no motivation or suggestion for modifying Sanhatjian to have the limitations of Claim 142. The records shows that the Examiner (Office Action mailed September 7, 2006 and previous Office Actions) also rely on In re Leshin to supply Sanhatjian's deficiency with regard to applicant's claimed polyurethane. Reliance on In Re Leshin with respect to claim 142 is not appropriate. In Leshin, the material in question, plastic, was selected for use in the claimed device based on its known suitability for the applicant's intended purpose. Only the applicants have recognized the suitability of polyurethane having a glass transition temperature above a storage temperature of a sheath material. No prior art cited in the record provides any sort of recognition that applicant's claimed polymer would be suitable as a sheath material.

Applicant submits that the rejection of Claim 142 under 35 U.S.C. § 103 is in error and respectfully request that the rejection be reversed.

Issue 2

The Examiner has rejected Claim 143 as being obvious over Sanhatjian. The rejection is improper because the cited art does not disclose at least the following limitation of Claim 143: “the polymeric material comprises a polyurethane having a non-polar soft segment wherein the non-polar soft segment is selected from hydrocarbons, silicones, fluorosilicones, or their mixtures” and because there is no motivation or suggestion in the record to modify the cited art to include the limitation.

The record shows that the Examiner acknowledges (Office action mailed September 7, 2006) that Sanhatjian does not disclose the above-mentioned limitation of Claim 143. However, the Examiner asserts that “it is common knowledge in the chemical art to modify the non-polar segments in order to provide properties such as flexibility and bendability which are desired in the medical arts” and concludes that “it would have been obvious to modify any medically acceptable material to the essential properties, which are desired.” These assertions in the record are without merit for the following reasons.

The Examiner is required to identify the differences between the invention as claimed and then reason why one of ordinary skill in the art would modify the prior art to get to applicant’s claimed invention. Graham v. John Deere Co., 383 U.S. 1, 17-18 (1965) While skilled artisans know to modify polymers to change their properties, obviousness in this case requires them to know which modifications to make and by how much. This remains beyond the capacity of skilled artisans. With respect to the capacity of skilled artisans, the Examiner is making factual assumptions which is the same as taking official notice of those facts. Indeed, M.P.E.P. § 2144.03 states that “the rationale for supporting an obviousness rejection may be based on common knowledge in the art or ‘well-known’ prior art” and the “examiner may take official notice of facts outside of the record which are *capable of instant and unquestionable demonstration* as being ‘well-known’ in the art.”

The record does not contain any references showing that one of ordinary skill in the art, possessing the teachings of Sanhatjian, would immediately envision applicant’s claimed

invention. Additionally, the record does not contain evidence that non-polar segments provide flexibility and bendability to polymers. The Response to Arguments section of the Office Action mailed May 4, 2005 states in reference to applicant's claimed polymer "the material is known as the prior art shows the material used in the food industry." However, no specific material used in the food industry is cited. The record does not include a reference that discloses a polymeric material with "a polyurethane having a non-polar soft segment" in which "the non-polar soft segment is selected from hydrocarbons, silicones, fluorosilicones, or their mixtures."

To establish a *prima facie* case of obviousness, the Examiner is required to find some motivation or suggestion to make the claimed invention in light of the prior art teachings. MPEP 2143. Sanhatjian discloses the use of polyurethane and a commercial medical polyurethane, Tecoflex™ (which does not have the above-mentioned claim limitations of Claim 143). However, there is no suggestion or motivation within Sanhatjian to use a polyurethane having the above-mentioned claim limitations of Claim 143. MPEP § 2144.08 (4) requires the Examiner to determine whether one of ordinary skill in the relevant art would have been motivated to make the claimed invention as a whole, i.e., to select the claimed species or subgenus from the disclosed prior art genus. Sanhatjian discloses the use of a genus, polyurethane, and a species, Tecoflex™, which does not have the limitations of Claim 143. There is no evidence in the record that one of ordinary skill in the art of polymers and medical devices would have been motivated to select a polymer having the limitations of Claim 143 for use in a sheath.

Many materials provide properties such as flexibility and bendability (such as Tecoflex™) other than the polyurethane recited by Claim 143. There is no motivation in the record to select applicant's claimed polyurethanes over many other polymers which could be selected that have bendability and flexibility. The motivation provided in the record that "it would have been obvious to modify any medically acceptable material to the essential properties" is merely an "obvious to try" rationale in support of an obviousness rejection. According to MPEP § 2145, "obvious to try" is not the standard under section 103.

In addition, the Office Action mailed September 7, 2006 relies on In re Leshin to supply Sanhatjian's deficiency with regard to applicant's claimed polyurethane. As discussed

above, In Leshin, the material in question, plastic, was selected for use in the claimed device based on its known suitability for the applicant's intended purpose. Based on the record, such is clearly not the case. Only the applicants have recognized the suitability of the claimed polyurethane as a sheath material. The only prior art cited in the record against claim 143 does not provide this recognition since the polyurethane disclosed by Sanhatjian is a different polymer with different properties. Other than applicant's specification and claims, there is nothing in the record concerning applicant's claimed polymer. Since there is nothing in the record about the claimed polymer, there is certainly nothing about its suitability for applicant's intended purpose.

Applicant submits that the rejection of Claim 143 under 35 U.S.C. § 103 is in error and respectfully request that the rejection be reversed.

Issue 3

The Examiner has rejected Claim 147 as being obvious over Sanhatjian. The rejections are improper because the cited art does not disclose at least the following limitation of Claim 147: "the balloon material, sheath material, or both comprise a therapeutic substance contacting surface" and because there is no motivation or suggestion in the record to modify the cited art to include the limitation.

Claim 147 depends from Claim 146 which recites "the balloon material, sheath material, or both comprise a therapeutic substance contacting surface." Claim 147 recites the limitation "the therapeutic substance contacting surface contacts a coating comprising a main-group-element oxide." Sanhatjian does not teach or suggest the limitations of Claim 147. The record does not provide a suggestion or motivation, either in a prior art reference or in the knowledge generally available to one of ordinary skill in the art, to supply Sanhatjian's deficiency with regard to the limitations of Claim 147. Therefore, a *prima facie* case of obviousness has not been made.

Applicant submits that the rejection of Claim 147 under 35 U.S.C. § 103 is in error and respectfully request that the rejection be reversed.

II. Conclusion

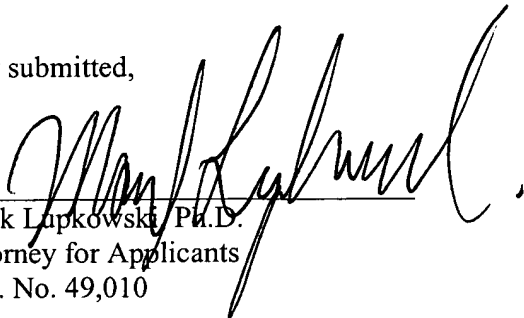
Application No. 09/406,473
Appeal Brief
March 20, 2007

For the foregoing reasons, the rejection of Claims 142, 143, and 147 as being unpatentable under 35 U.S.C. 103 over Sanhatjian is in error, and the Board is respectfully requested to reverse the rejection.

Respectfully submitted,

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Claims Appendix

139. A sheath comprising a hollow body, the sheath being adapted to removably cover at least part of an implantable medical device that carries a therapeutic substance, wherein the sheath material prevents the therapeutic substance from significantly absorbing into the sheath, wherein the sheath material has an oxygen transmission rate of not more than 200 cc/100 in² for 1 mil per 24 hours at 73°F, 75% relative humidity and 1 atmosphere, and wherein the sheath material has a water vapor transmission rate of not more than 20 gm/100 in² for 1 mil per 24 hrs. 100°F 90% relative humidity and 1 atm.
140. The sheath of Claim 139 wherein the implantable object is a balloon integrated with a catheter.
141. The sheath of Claims 139-140 wherein the balloon material, sheath material, or both comprise the same or different polymeric material.
142. The sheath of Claim 141 wherein the polymeric material comprises a polyurethane having a glass transition temperature above a storage temperature.
143. The sheath of Claim 141 wherein the polymeric material comprises a polyurethane having a non-polar soft segment wherein the non-polar soft segment is selected from hydrocarbons, silicones, fluorosilicones, or their mixtures.
144. The sheath of Claim 141 wherein the implantable object comprises a stent.
145. The sheath of Claim 141 wherein the polymeric material comprises polyolefins, polyurethanes, derivatives of cellulose, polyesters, polyamides, poly(hexamethylene isophthalamide/terephthalamide), poly(ethylene terephthalate-co-p-oxybenzoate), poly(hydroxy amide ethers), polyacrylates, polyacrylonitrile, acrylonitrile/styrene copolymer, rubber-modified acrylonitrile/acrylate copolymer, poly(methyl methacrylate), liquid crystal polymers, poly(phenylene sulfide), polystyrenes, polycarbonates, poly(vinyl alcohols), poly(ethylene-vinyl alcohol), epoxies composed of bisphenol A based diepoxides with amine cure, aliphatic polyketones, polysulfones, poly(ester-sulfone), poly(urethane-sulfone), poly(carbonate-sulfone), poly(3-hydroxyoxetane), poly(amino ethers), gelatin, amylose, parylene-C, parylene-D, parylene-N, or their mixtures.

146. The sheath of Claim 141 wherein the balloon material, sheath material, or both comprise a therapeutic substance contacting surface.
147. The sheath of Claim 146 wherein the therapeutic substance contacting surface contacts a coating comprising a main-group-element oxide.
148. The sheath of Claim 147 wherein the main-group-element oxide comprises a silicon oxide, metal oxide, or a mixture of a silicon and a metal oxide.
149. The sheath of Claim 141 wherein the sheath material lines at least a portion of the inner surface of the sheath in a layer.
150. The sheath of Claim 149 wherein the implantable object is disposed within the sheath for transportation or storage.
151. The sheath of Claim 141 wherein the material is selected from:
 - polyurethane having a glass transition temperature above a storage temperature;
 - polyurethane having a non-polar soft segment wherein the non-polar soft segment is at least one of hydrocarbons, silicones, fluorosilicones, or mixtures thereof;
 - at least one cellulose derivative selected from cellulose acetate having a degree of substitution greater than about 0.8, ethyl cellulose, cellulose nitrate, cellulose acetate butyrate, methyl cellulose, or mixtures thereof;
 - sulfonated polymers;
 - fluorinated polymers;
 - carbide compounds;
 - nitride compounds;
 - a polyolefin that is at least one of polyethylenes, poly(vinyl chloride), poly(vinylidene chloride), poly(vinyl fluoride), poly(vinylidene fluoride), poly(tetrafluoroethylene), poly(chlorotrifluoroethylene), or mixtures thereof;
 - a polyester that is at least one of poly(ethylene terephthalate), poly(ethylene 2,6-naphthalene dicarboxylate), poly(butylene terephthalate), or mixtures thereof;

a polyamide that is at least one of nylon-6; nylon-6,6; nylon-6,9; nylon-6,10; aromatic nylon; or mixtures thereof; and

and any combinations thereof.

152. The method of Claim 151 wherein the polymeric material comprises a polyurethane having a glass transition temperature above a storage temperature.
153. The method of Claim 151 wherein the polymeric material comprises a polyurethane having a non-polar soft segment wherein the non-polar soft segment is selected from hydrocarbons, silicones, fluorosilicones, or their mixtures.
154. The method of Claim 151 wherein the implantable object comprises a stent.

Application No. 09/406,473
Appeal Brief
March 20, 2007

Evidence Appendix

An Investigation of the Fatigue Induced Failure Modes of Fiber/Elastomer Composites as Bearing Surfaces in Total Hip Joint Prosthesis

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M. LaBerge, Ph.D., Associate Professor of Bioengineering

Goal Statement

This research represents a fundamental study of the failure mechanisms observed in fiber reinforced elastomeric bearing surfaces. The goal of this research is to develop a better fundamental understanding of all complex modes of failure in bearing surfaces which can then be used to design new materials with sufficient longevity for use in total joint replacements or other bearing applications.

Abstract

It is proposed to use elastomeric composites as artificial joint bearing surfaces to increase their fluid film lubrication and therefore reduce the number of failures which have been attributed to wear mechanisms. The fluid generated between the surfaces supports part of the load and prevents direct contact of the bearing surfaces. In order to obtain optimum elastohydrodynamic lubrication, elastomeric materials are selected based on their static mechanical properties such as modulus of elasticity. However, the observed permanent deformation and failure of model elastomeric composites has been associated with debonding between fibers and matrix. It is hypothesized that the operating conditions such as frequency (velocity) and temperature at the contact between the elastomeric composite and the reciprocating counter part may contribute significantly to the failure of the elastomer. The mechanical properties of elastomers as of other polymers are highly dependent on strain rate, frequency, and temperature. Polyurethane thermoplastic elastomers of three different hardnesses (85A, 93A, 100A) were characterized with dynamic mechanical thermal analysis. The coefficient of friction between these materials and metal was measured using reciprocating motion. Data showed that the lubrication mode of a metal-elastomer contact in tribological conditions observed in artificial joints is highly dependent on temperature and frequency. In this respect, dynamic mechanical analysis can be used in the selection of an optimal elastomeric material for reciprocating bearing conditions.

Introduction

Low modulus elastomeric coatings were proposed and investigated by several authors as a method to improve the tribological performance of bearings (Unsworth et al., 1987; Dowson et al., 1991). By deforming under pressure, elastomeric layers enhance lubrication by the formation of a fluid film through elastohydrodynamic and micro-elastohydrodynamic lubrication (Dowson and Jin, 1986). This occurs when the asperities of these materials flatten due to local pressure perturbations. However, when the fluid film breaks down, adhesive friction increases in presence of smoother elastomeric materials (Fuller and Tabor, 1975). Lower modulus materials provide more effective micro-elastohydrodynamic lubrication but fail due to an increase in shear strain. Dowson et al. (1991) suggested that the elastic modulus should not be lower than necessary to provide effective micro-elastohydrodynamic lubrication. However, these authors did not address the fundamental effect of temperature (heat) as experienced in tribological contacts and operating conditions on the dynamic mechanical properties of polymers and elastomers and therefore, on the lubrication mechanism and subsequent failure of the elastomers.

Research on the use of LME as bearing surfaces done thus far has focused on the evaluating experimentally and theoretically the tribological behavior of the elastomer layer. However, a more in-depth understanding of the material properties of elastomers is necessary to predict the behavior of these bearings in the complex environment of the body. Two variables which can have a profound effect on the mechanical properties, such as the modulus of elastomers, and have not been considered to date in the available literature are temperature and frequency. The objective of this research was to:

" characterize the effects of temperature and frequency on the modulus of a selected set of elastomers, and to relate these parameters to both, theoretical and experimental tribological results. "

The long term goal of this research is to develop a better fundamental understanding of all of the complex modes of failure in bearing surfaces which can then be used to design new materials with sufficient longevity for use in a total

joint replacement or industrial bearings. The following specific aims were addressed this year in order to reach this goal:

- to characterize the thermal and thermo-mechanical material properties of a selected set of polyurethane elastomers,
- to evaluate the theoretical tribological properties of the polyurethane bearing surface based on the results of the materials characterization, and
- to compare the experimental results of the tribological characterization of the elastomeric layers as a function of frequency and temperature with the theoretical tribological evaluation.

Materials & Methods

In order to reach the above goals, the project was divided into four phases: 1) fabrication of testing samples, 2) dynamic mechanical thermal analysis of the test samples, and 3) tribological analysis of the contact elastomer-metal as a function of frequency and temperature.

1. Sample Fabrication

Three grades of a low modulus medical grade aliphatic polyether-based polyurethane elastomer (Tecoflex® SG 85A, 93A, 100A by Thermedics Inc., Woburn, MA) were used to as test materials. Tecoflex® resins are reaction products synthesized from methylene bis(cyclohexyl) diisocyanate [HMDI], poly(tetramethylene ether glycol) [PTMEG], and 1,4 butane diol chain extender. Based on our previous results showing that solution casting did not allow a uniform thickness layer greater than 1 mm to be obtained, the test samples were compression molded. Prior to fabrication, the hydroscopic pellets were dried in a vacuum oven at approximately 75°C for at least three hours to remove any water content and stored in a desiccator until the time of fabrication. The pellets were placed in a 2.38 mm thick polytetrafluoroethylene mold (8.89 cm X 11.43 cm) with polytetrafluoroethylene release ply (Teflon®/Wrightlon 5900, Airtech International, Carson, CA) placed between the elastomer pellets and the metal plates to prevent the elastomer from adhering to the plates. The pellets were then heated in a Carver laboratory press (Model C, Fred S. Carver Inc., Wabash, IN) for 25 minutes, up to 350°F, under a constant pressure of 3000 lbs and cooled down to room temperature using the cooling system on the press.

Samples were then sectioned for the dynamic mechanical thermal analysis (DMTA) and tribological analysis. Thermocouples (0.005 inch diameter Type T, Omega, Inc.) were embedded into the elastomer (1 mm from surface) in several positions to measure the frictional heat generated as a function of time.

2. Material Property Characterization

Dynamic mechanical thermal analysis (DMTA) is being used in this project to characterize the thermo-mechanical properties of the elastomer, such as the glass transition temperature, the loss modulus, and storage modulus. These material parameters are used in studying the fatigue failure of the Tecoflex® utilizing the principle of time-temperature super positioning.

The time-temperature superposition principle can be very useful in studying polymers. It allows for the extrapolation of the transition behavior to frequencies beyond the instruments measurement range and separates the effects of time and temperature. To achieve this superposition, DMTA curves obtained at various frequencies during pseudo-isothermal scans are shifted to some arbitrary reference temperature by a horizontal shift factor, aT , to produce a composite "master" curve covering a wider frequency range.

The DMTA experiments (Seiko DMS 210) were run on the various solution grades of the Tecoflex® including 80A, 85A, 93A, 100A, and 60D where the A and the D designate the Shore hardness scale. The experiments were run from -100°C to 100°C at 0.05°C/min. using eight frequencies ranging from 0.02 to 10 Hz. Data obtained with DMTA were used to theoretically predict the lubrication modes as will be described below.

3. Tribological Analysis

The effect of reciprocating motion and friction between mirror polished cylindrical cobalt-chrome alloy (ASTM F-75) bearings and flat elastomeric sheets were evaluated under mixed and fluid film lubrication conditions using a reciprocating pin-on-plate friction table. This 3-station pin-on-plate friction table simulates the line contact as in a typical artificial knee joint. Strain gauges are used to determine the friction force. Friction tests were conducted at various operating speeds simulating normal joint motion to evaluate the effect of operating frequency on the lubrication mode. A multi-channel data acquisition system was designed using LabView™ (National Instruments, TX) to measure T below the line contact and friction as a function of time. A tribological model previously

developed by the investigators to study the effect of material stiffness and lubricant viscosity on artificial joint lubrication was used to generate calibration curves (friction versus Sommerfeld number). Tests were conducted at room temperature (RT), 37°C (body temperature), and 45°C (temperature of an inflamed joint often observed after joint replacement).

The low modulus materials were secured to the reciprocating table and served as the lower bearing surface. The total travel distance was 124 mm/cycle or 62 mm in each direction. The velocity of the platform was controlled by a speed dial adjacent to the system and was monitored by a linear variable displacement transducer (Model 3000 HR, S/N 320, Lucas Schaevitz, Pennsauken, NJ). Tests were conducted at 1Hz and 2Hz simulating slow and fast walking patterns under a contact stress of 2 MPa (within physiological stresses observed in human joints). 20 ml of lubricant was placed on the bearing surface. Glycerin (Humco Laboratory, Texarkana, TX) was chosen as lubricant because it is a Newtonian fluid and therefore independent of the shear rate. Glycerin was diluted with distilled water to obtain an 86% solution. The concentration chosen for this study corresponded to the apparent viscosity of degenerated synovial fluid at low shear rates ($\sim 1 \text{ s}^{-1}$) which was 100 cP, as given by Davies and Palfrey (1969). According to Miner and Dalton (1953), an 86% glycerin solution has an absolute viscosity of 100 cP at 25°C. The upper bearing surface was then lowered on top of the lower bearing surface and the machine was turned on. Resistance to sliding was measured by the force transducers. The voltage output from the transducers was amplified and used to determine the force required to hold the jig in place while the reciprocating plate moved. By use of Amonton's law, the coefficient of friction could be determined. All tests were conducted in an environmental chamber. A total of six tests were conducted for each set of experimental conditions (frequency and temperature) for each grade for up to 50,000 cycles.

A statistical analysis was performed using a general linear model to compare the effect of frequency and grade on dynamic mechanical properties as well as the effect of experimental conditions on optimization of lubrication modes.

Results and Discussion

Material Characterization

The DMTA curves obtained for the different grades of Tecoflex® illustrate the effects of both the soft and the hard segments in its composition. The value of $\tan \delta$, the glass transition temperature (T_g), and the loss and storage modulus were compared as a function of grade, frequency, and temperature. The purpose of evaluating T_g was to understand the effects of frequency on the glass transition temperature and characterize the difference in the material behavior from both a thermo-mechanical and tribological view both above and below this transition.

The soft 85A grade of elastomer exhibited two primary transitions on the $\tan \delta$ curve. The first peak on the 1 Hz DMTA curve occurred at approximately, -57°C and the second, more prominent peak on the $\tan \delta$ curve was observed at approximately +28°C. This two peak behavior is representative of the two glass transition temperatures of both the hard and the soft segments and has been seen in other two phase microstructure polymer systems (Encyclopedia of Polymer Science, 1989). The -57°C representing the transition of the soft polyether segments and the +28°C that of the T_g for the hard urethane segments. An additional indication of transitions at both of the temperatures is the change in slope of both the storage and loss modulus curves. The 93A which was the intermediate polyurethane hardness grade evaluated demonstrated only one prominent peak above room temperature which would represent the glass transition temperature of the hard urethane segments. However, both the loss and storage modulus as well as the $\tan \delta$ curves indicate an additional transition at around -50°C, which occurs close to the glass transition temperature of the soft segments. The 100A grade of Tecoflex® polyurethane, the hardest grade evaluated, also showed its most distinct peak at approximately 50°C which again corresponds to the hard segment transition. A slight change in slope of both the storage and loss modulus curves at around -50°C was also indicative of a transition in soft segments of the polyurethane polymer system. The DMTA's from both the 93A and the 100A suggest a stronger influence of the hard segments in comparison to the 85A because of the presence of one prominent transition instead of the two. However, the soft segments are present in both elastomers and their interaction with the hard domains will effect any of the elastomer's material properties. At the lower content of the hard segments present in the elastomer, as with the 85A grade, the structure of the elastomer can be described as disordered hard segments distributed within a matrix of the soft polyether segments. As the amount of hard segments increases, in the case of the 93A and 100A elastomeric grades, the more hard segments can aggregate and organize into lamellar hard microdomains. The transition between the two occurs at a critical segment lengths as defined by Koberstein et al. (1992) and Lelah et al. (1986).

The mean Tg results as a function of frequency obtained from the DMTA experiments are summarized in Table 1. The most profound effect of frequency was on the 85A grade of Tecoflex® elastomer at both transition peaks. A statistical analysis of the 85A data which compared the mean recorded Tg values using ANOVA ($\alpha=0.05$) was done and the glass transition temperature at all eight of the frequencies were statistically significantly different from each other at both the hard and soft segment glass transition peaks. For both the 93A and 100A elastomer grades, the effect of frequency on the Tg was not as clearly defined between each pair of frequencies as with the 85A. Overall, only the glass transition temperatures at both 0.1 and 0.02 Hz for each of these two grades of polyurethane were determined to be statistically significantly different from all of the other frequencies and each other. The standard deviations of the Tg values obtained at the higher frequencies for the 93A and 100A elastomer grades overlapped each other.

Table 1 Mean Values and Standard Deviation for the Glass Transition Temperature (°C)

Grade	10 Hz	5 Hz	2 Hz	1 Hz	0.5 Hz	0.2 Hz	0.1 Hz	0.02 Hz
85A Mean	-45.68	-49.76	-53.68	-57.52	-59.90	-63.68	-67.00	-69.10
Std Dev.	4.13	6.88	5.51	4.40	4.45	3.88	3.36	4.62
85A Mean	53.54	47.10	37.00	28.44	22.92	15.46	8.06	1.94
Std Dev.	9.84	11.26	13.61	14.03	14.16	15.46	3.65	1.90
93A	41.98	37.40	32.22	29.12	25.78	22.42	18.84	14.72
Std Dev.	3.91	3.14	3.01	1.96	2.98	2.66	4.34	1.21
100A	53.26	50.82	47.36	44.92	42.52	40.14	38.56	32.68
Std Dev.	0.92	0.95	1.53	1.53	1.49	2.83	2.46	4.07

Dynamic mechanical analysis results showed that at the test frequencies used to perform the frictional analysis, the elastomeric material is significantly behaving differently. An increase in modulus is observed for the three grades studied, 85A, 93A, and 100A, as a function of frequency increase (Figure 1) and temperature observed in the body (37°C is body temperature and 45°C is reported temperature between articulating orthopaedic surfaces (metal-polyethylene) (Figure 2). These results indicate that the use of a single value as representative of the mechanical properties of the elastomer in the prediction of lubrication mechanisms for different test frequencies is not appropriate since the material behaves differently as a function of this parameter.

For each grade of elastomer studied, the modulus decreased as a function of temperature as the elastomer began to soften, except for the case of the 85A at 45°C (Table 2). This apparent discrepancy of the 85A grade at 45°C can be attribute to the large standard deviation. One of the five modulus measurements was double the modulus of the others. Since no specific reason could be identified why this test should be disregarded, this result was kept in the data for analysis purposes. Without this modulus value of 14.17 MPa, the 85A at 45°C would average 7.74 MPa instead of 9.17 MPa. The statistical analysis of the 85A indentation modulus data indicated that all of the calculated mean modulus values as a function of temperature were statistically significantly similar to each other when compared using ANOVA ($\alpha = 0.05$). Similar results were obtained from the statistical analysis of the other two grades.

Table 2. Comparison of TMA as a function of temperature and elastomeric grade.

Tecoflex® Sample	Modulus @ 25°C (MPa)	Modulus @ 37°C (MPa)	Modulus @ 45°C (MPa)
85A	7.80 ± 0.88	7.36 ± 1.12	9.17 ± 3.32
93A	16.03 ± 1.85	14.52 ± 3.11	12.75 ± 1.88
100A	12.78 ± 2.95	10.71 ± 2.24	8.12 ± 2.02

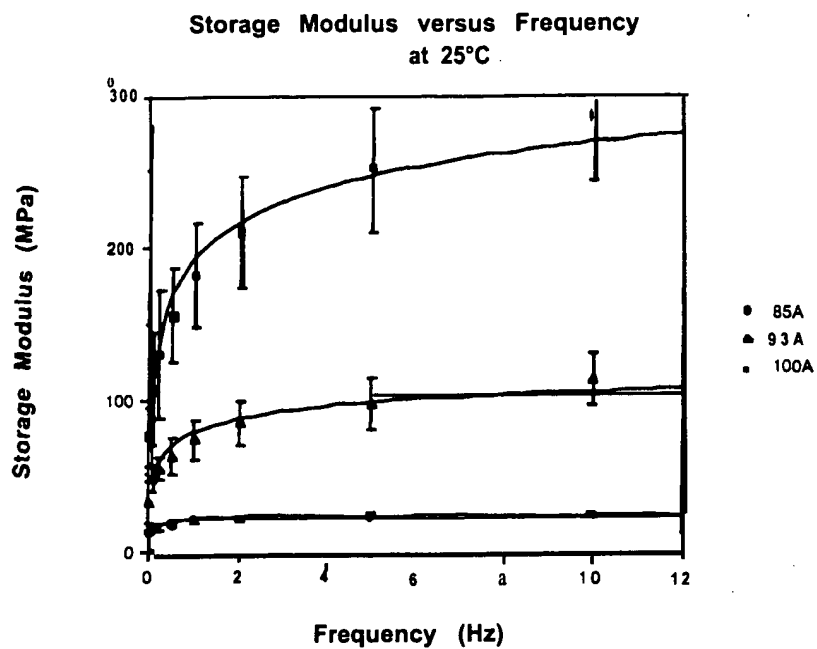


Figure 1. Effect of the frequency on the storage modulus of different grades (hardnesses) of Tecoflex.

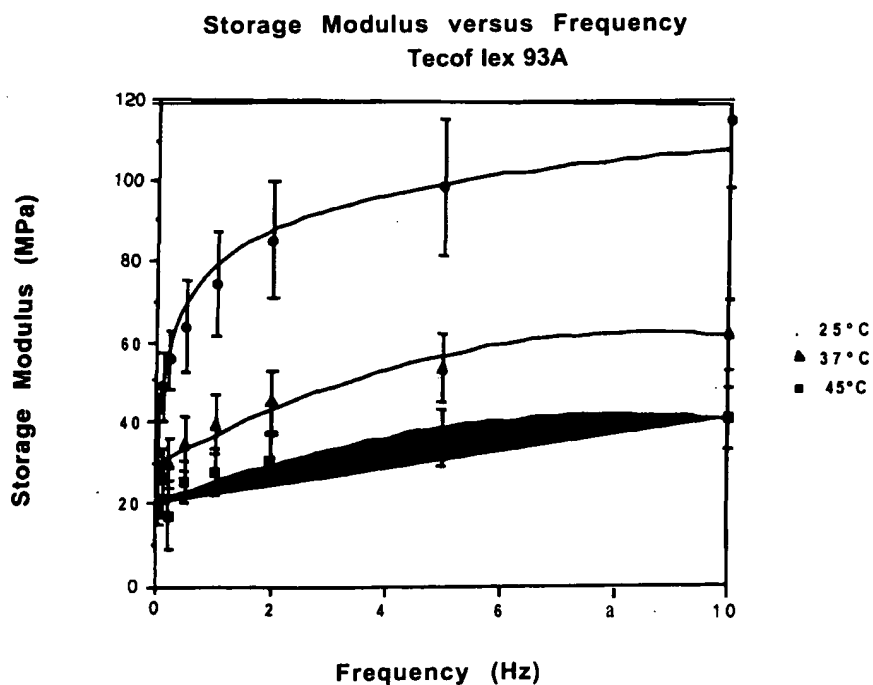


Figure 2. Effect of frequency on the storage modulus of Tecoflex SG93A for different operating temperatures.

Elastomers are profoundly influenced by both temperature and frequency as demonstrated in this material characterization section. The most profound influence of these parameters occur in the stiffer grades of elastomer as the hard segment content is increased. In the literature, however, the importance of these visco-elastic properties have often not been considered in the evaluation, such as in the case of the polyurethane elastomers being considered for cushion bearing joint replacements. To date, in the available literature, all of the tribological testing reported was done at room temperature. The body temperature is approximately 12°C higher than room temperature and the mechanical properties of an elastomer have been shown in this analysis to change significantly as a function of temperature over this range. As a result, this author believes the tribological properties of cushion bearing joints will be profoundly influenced by the combination of temperatures and frequency.

Tribological Analysis

The reason for analyzing the lubrication regimes which were present experimentally and would be predicted theoretically was to gain an understanding of how much interaction existed between the bearing surfaces. The lubrication regime is based on the calculation of the lambda ratio (λ) which is based on both the central film thickness as well as the surface roughness. When the λ is less than 1, the boundary lubrication regime dominates and the extent of surface contact is high. For a λ of 3 and higher, the elastohydrodynamic lubrication regime theory predicts no contact of the bearing surfaces will occur. In the mixed lubrication regime, $3 \leq \lambda \leq 1$, intermittent surface interaction does occur.

The contact stress analysis, necessary to generate the λ ratio, was done for each of the elastomer grades as a function of frequency and temperature. The effects of frequency and temperature have been shown to profoundly impact the resulting stress values for all of the grades of elastomer. The basic trend is as temperature increases, the stress is reduced. The reduced stress is the direct result of the lower modulus values as the material is able to deform.

The 93A grade of elastomer was selected as the candidate material for tribological analysis because it was the intermediate grade evaluated in the material characterization portion of this research. The friction testing was done at room temperature ($\approx 22^\circ\text{C}$), 37°C , and 45°C and at the frequencies of 1 and 2 Hz. The coefficient of friction in general decreased as a function of time for all of the experimental conditions with 100 cycles being typically the highest friction and 5000 cycles being the lowest. Statistical analysis using ANOVA ($\alpha=0.05$) was done initially to study the effects of the number of cycles. The only statistical differences observed in the friction values as a function of time occurred between 100 and 5000 cycles. Therefore, for the statistical analysis of the effects of frequency and temperature, comparisons were only made with the 100 and 5000 cycles friction data. Overall, the experimental friction values measured during the course of the testing ranged from 0.03 to 0.1 depending on the frequency and temperature of the testing. This trend has also been observed by other authors and is the result of theory not including EHL effects (Auger et al., 1990; Auger et al., 1993; Caravia et al., 1993; Graham, 1994). The reason for this difference in the theory is that it is strictly based on the ratio of viscosity, velocity, contact area, applied load, and central film thickness. In these cases, the only variables which are changing are the velocity, contact area, and the central film thickness. The equation does not consider EHL or micro-EHL effects.

The minimum fluid film thickness and the lambda ratio results for the experimental portion of the tribological characterization are presented in Table 3. Based on the results for the lambda ratio, the lubrication mode for the experimental tribo-system was identified. The modulus values used for the calculations were from the DMTA storage modulus results for the 93A grade of elastomer at the frequencies 1 and 2 Hz. The basic trend observed was the higher the storage modulus, which corresponded with the higher frequency measured by the DMTA, the lower the minimum fluid film thickness and the lambda ratio. This phenomena can be explained by understanding the relationship between the modulus and the resulting fluid film thickness generated. The higher modulus elastomers, as in the case of the storage modulus values at the higher frequencies, are stiffer by nature and are not able to deform as much under the pressure wedge generated in the lubricant as a function of velocity. As a result, thinner lubricating films are created which in turn, depending on the surface roughness, have lower λ ratio values. With the rise in temperature, however, a decrease in the film thickness and the lambda ratio was observed, opposite to the expected increase in both the minimum fluid film thickness and the λ ratio. The reason for this decrease was attributed to the decrease in the viscosity of the glycerin. At 25°C , the viscosity of the glycerin is 945 cP which decreases dramatically at 37°C to 500 cP and at 45°C to 300 cP. The decrease is not as dramatic as if the stiffer 25°C DMTA storage modulus of the 93A elastomer were to be used in the calculations for the fluid film thickness and the lambda ratio at both 37°C and 45°C . Based on the equation for calculating the minimum fluid film thickness in which the modulus is in the denominator, the lower storage modulus values measured by the DMTA

compensate for the decrease in the viscosity of the lubricant with temperature and allowed for greater minimum fluid film thickness and a higher λ ratio (Figures 3, 4, and 5).

Table 3. Experimental Lubrication Analysis Results

Velocity (mm/sec)	Frequency (Hz)	Minimum Film Thickness			Lambda Ratio		
		25°C	37°C	45°C	25°C	37°C	45°C
10	2	1.34	1.17	1.00	1.01	0.89	0.76
10	1	1.41	1.23	1.05	1.07	0.94	0.79
30	2	2.59	2.27	1.93	1.96	1.72	1.46
30	1	2.73	2.39	2.02	2.07	1.81	1.53
60*	2	3.92	3.43	2.93	2.97	2.60	2.22

* The peak velocity for the 2 Hz experimental evaluation.

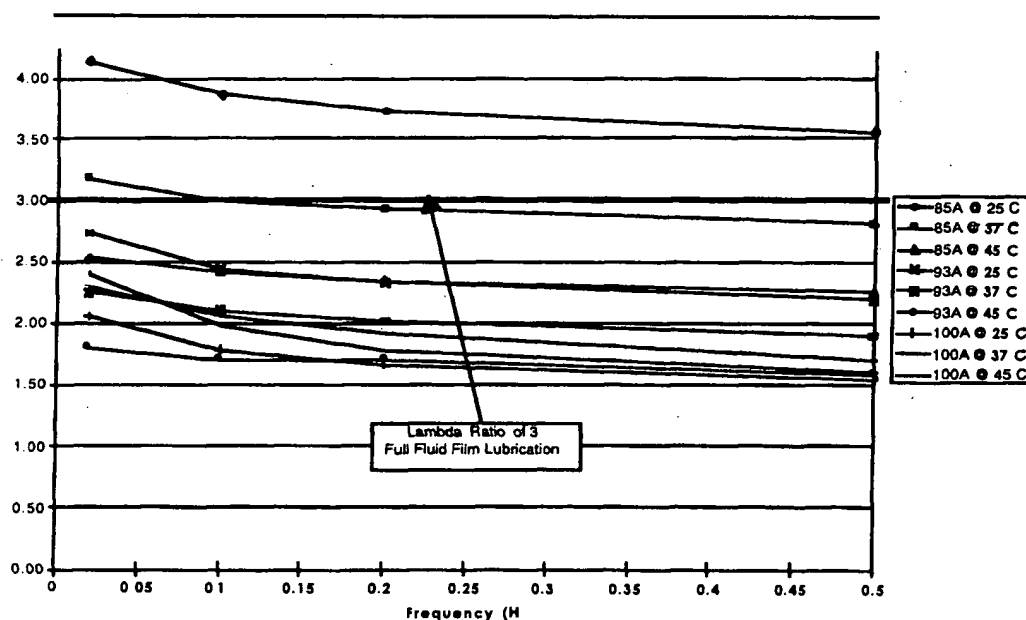


Figure 3. Lambda ratio based on the DMTA modulus results as a function of frequency at 30 mm/s at the lower frequencies.

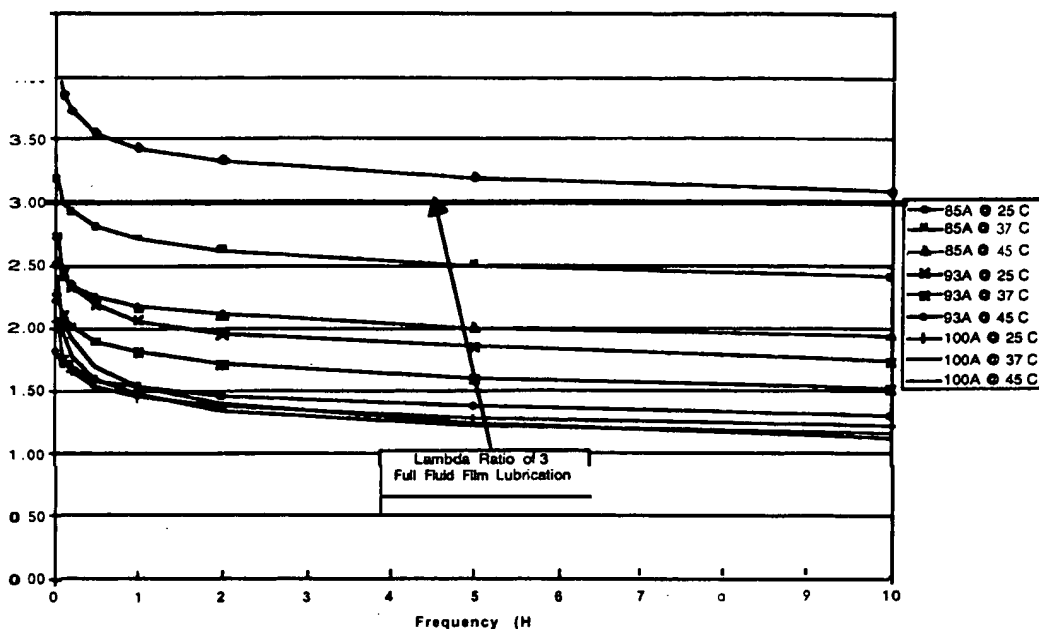


Figure 4. Lambda ratio based on the DMTA modulus results as a function of frequency at 30 mm/s.

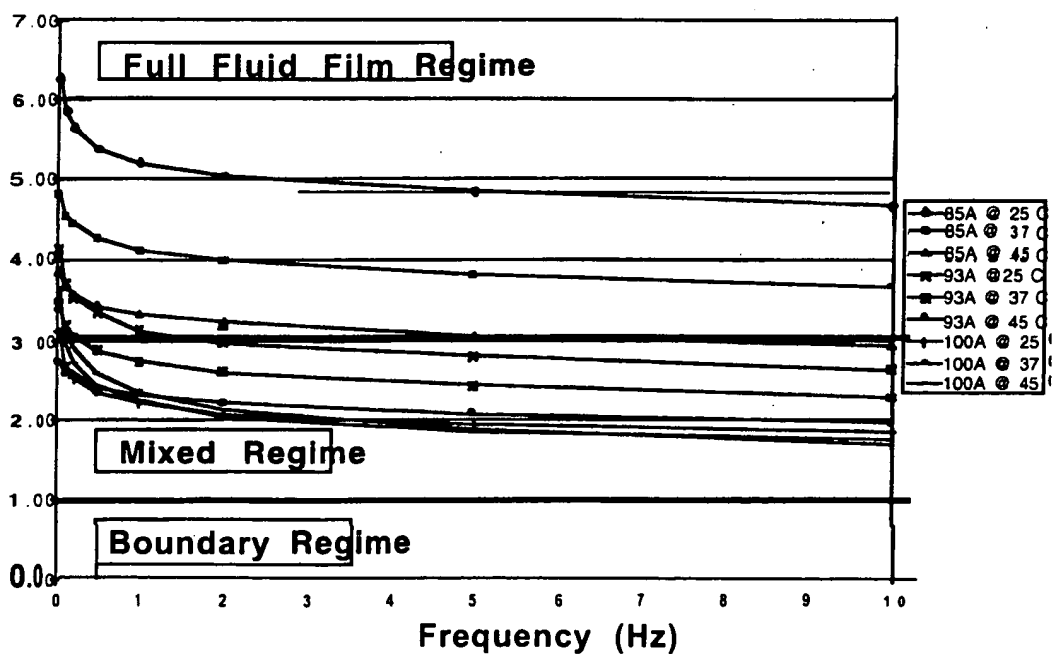


Figure 5. Lambda ratio based on the DMTA modulus results as a function of frequency at 60 mm/s

Results obtained with the tribological study suggest that for the grade SG93A, different lubrication mechanisms are observed with different environmental temperatures. Heat may have contributed to reduce the viscosity of the glycerin solution. For a 2.5° C increase, the viscosity of the glycerin solution would decrease by almost 10 cP

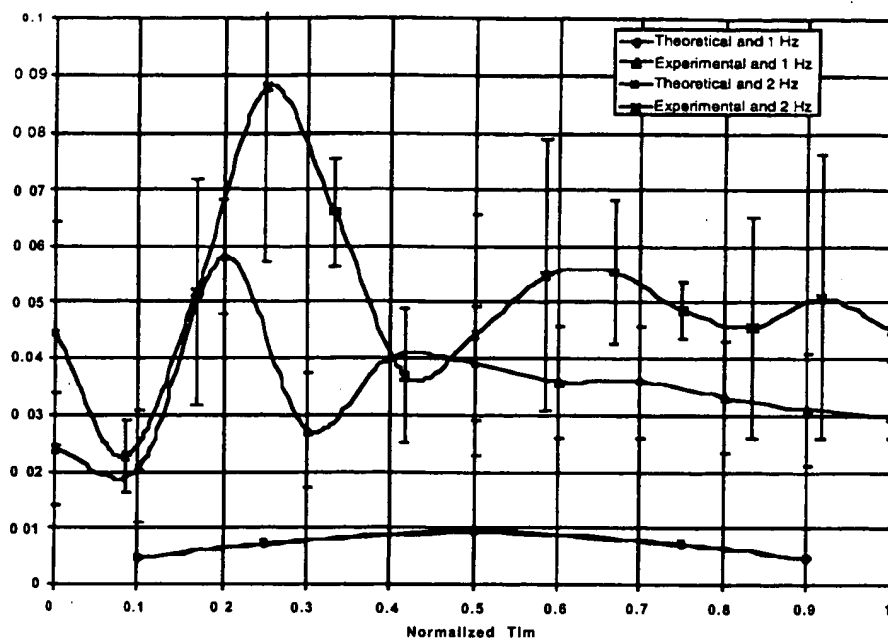


Figure 6. Theoretical and experimental coefficients of friction against normalized time at 37°C.

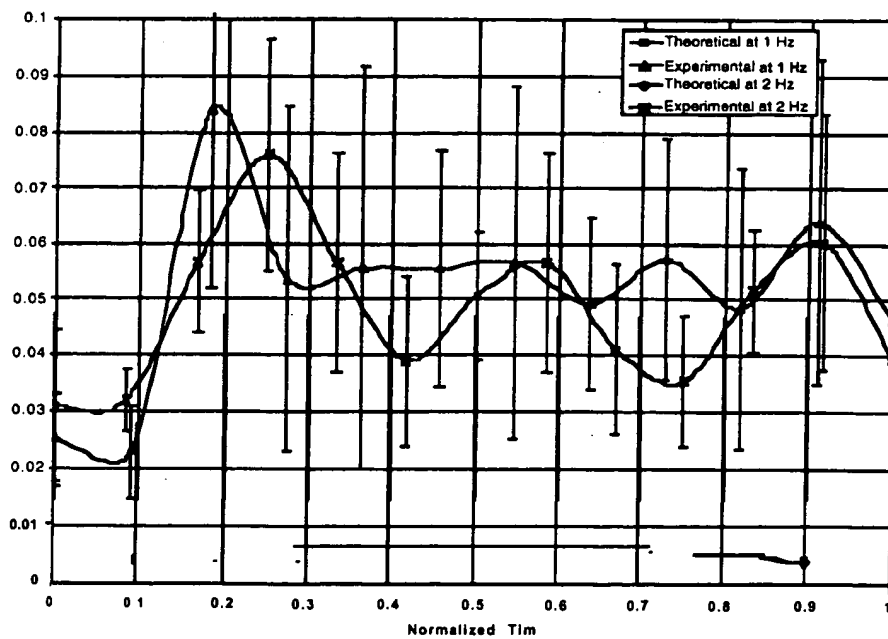


Figure 7. Theoretical and experimental coefficients of friction against normalized time at 45°C.

(Miner and Dalton, 1953). Therefore, temperature influences severely the viscosity of the lubricant and may have contributed to an increase in friction at higher temperature as shown with the theoretical curve (Figure 5). However, experimental results showed that overall fluid film lubrication is better observed at 37°C and 45°C than at room temperature. In these cases the experimental friction values exceeded the theoretical friction values (Figures 6 and 7).

Conclusions

Experimental studies conducted by Auger et al. (1992) and Caravia et al. (1993) have shown that the frictional behavior of elastomeric coatings was highly dependent on the modulus of elasticity of the coating at a low velocity (8 mm/sec). For the contact elastomer-metal, increasing the stiffness of elastomeric coatings increases the start up friction of the contact for boundary lubrication conditions (Caravia et al., 1993). Dowson et al. (1991) suggested that the elastic modulus should not be lower than necessary to provide effective micro-elastohydrodynamic lubrication. A further reduction in elastic modulus would therefore increase the shear strains above acceptable limits. The results obtained in this study also emphasize that the elastomer properties will be different at different frequencies which will contribute to possible failure of the material due to fatigue. Also, LME materials deform more easily thus enhancing micro-elastohydrodynamic lubrication, however, as stated previously the lower modulus layer cannot withstand the stresses and strains experienced under physiological conditions.

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